Grain boundary misorientation angles and stress-induced voiding in oxide passivated copper interconnects

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Grain boundary misorientations were determined by electron backscattering diffraction for tantalum-encapsulated, copper interconnects which contained thermal-stress-induced voids. The misorientation angles at voided and unvoided line segments were analyzed for two differently heat treated sample types, which were not equally susceptibile to stress voiding. Unvoided line segments contained a larger percentage of low misorientation angle, lower diffusivity boundaries than regions adjacent to voids. In addition, the more void resistant sample type also contained an overall higher proportion of low misorientation angle boundaries than the sample type which exhibited more voiding. The data provide further support for the importance of local variations in microstructure, which control the kinetics of stress void formation and growth. © *1997 American Institute of Physics.* [S0003-6951(97)00510-X]

Local microstructural variations are an important factor contributing to stress-induced voiding in passivated aluminum alloy[1,2] and copper interconnects.[3,4] Recent research, which concentrated primarily on local texture measurements within lines, identified grains immediately adjacent to voids as weaker in ⟨111⟩ texture than unvoided regions of the same lines.[2–4] This weaker, local texture implies the presence of twist boundaries, rather than tilt-type grain boundary character. Twist boundaries exhibit greater average diffusivity in the film plane, which increases their susceptibility to void formation and growth. Further results from one of the studies[3,4] confirmed the general belief that overall line texture correlates with general stress voiding reliability in copper. This work showed that weaker line texture was associated with a much higher density of voids. Analogous results were seen for aluminum-based lines,[1] although some discrepancies exist.[2] This letter details further analysis of electron backscattering diffraction (EBSD) data acquired from voided versus unvoided sites in narrow passivated copper interconnects. These results are presented in the form of grain boundary misorientation angle distributions and are discussed in terms of local diffusivity. Although grain boundary structures have five degrees of freedom, these results are analyzed only in terms of minimum misorientation angles, while neglecting boundary plane orientations. This is still a useful representation of boundary structure in the case of textured films where the boundary plane is nearly normal to the film plane.

Stress-induced voiding in passivated copper interconnects has been examined as a function of linewidth,[5] heat treatment,[5] and crystallographic orientation.[3] Arrays of tantalum cladded copper lines, 0.75–2.0 μm wide and 0.5 μm thick, were deposited by electron beam evaporation. A trilayer, lift-off stack was processed to ensure that the evaporated lines were rectangular in cross section. The lines were passivated with 1.2 μm of plasma-enhanced chemical vapor deposited oxide at 240 °C. To briefly summarize the heat treatments, type 1 samples were annealed for 1 h at 400 °C, only after passivation deposition. Type 2 samples were annealed for 1 h at 450 °C immediately following copper lift-off *and* were subjected to the post-passivation anneal.

The passivation and overlying tantalum layers were removed by reactive ion etching in a CF_4 ambient to expose the copper surface for analysis. Measurements from focused ion beam images revealed monomodal grain size distributions for each sample type, and equivalent average grain diameters of approximately 0.5 μm. EBSD patterns were acquired within a scanning electron microscope, with details of the setup[6] described elsewhere. Diffraction patterns were collected from grains surrounding 19 voids in type 1 and type 2 samples. Orthogonal arrays of EBSD patterns were also collected in unvoided regions of 1 and 2 μm wide lines, as described in Ref. 3. The voided and unvoided data sets were analyzed independently of linewidth.

Grain boundary misorientations were determined using the methodology described by Warrington and Bufalini.[7] From the trace, Tr, of the rotation matrix between the orientations associated with two adjacent grains, the misorientation angle, β is calculated as

$$\beta = \cos^{-1}\left(\frac{Tr-1}{2}\right). \tag{1}$$

Misorientation angles were determined for adjacent pairs of patterns in both voided and unvoided regions. With this approach, it became clear which patterns were obtained from the same grain, and which pairs arose from two grains sepa-

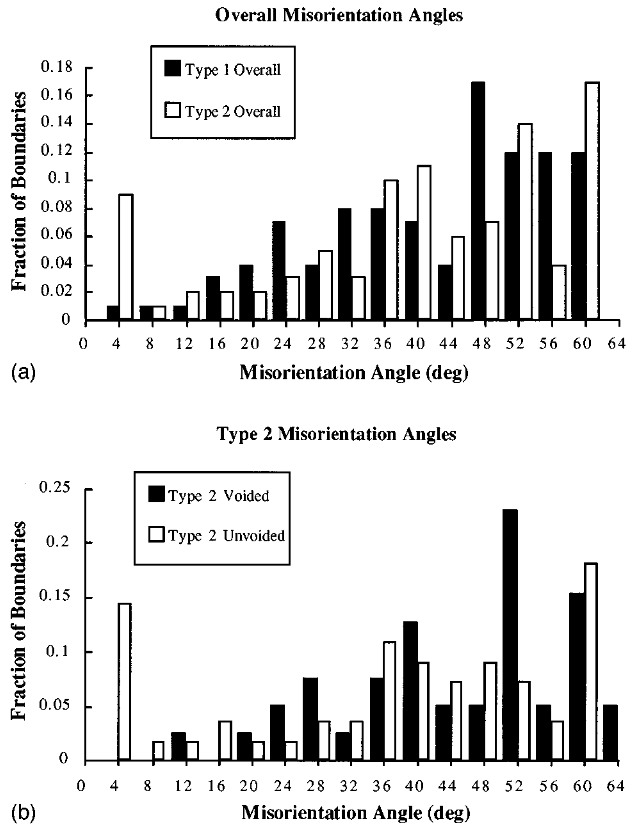

FIG. 1. Mackenzie plots for: (a) grain boundaries measured in both sample types, independent of voided or unvoided sites; (b) grain boundaries measured in type 2 samples, separated into voided and unvoided regions.

rated by a grain boundary. Misorientations of less than 2° were not considered to represent grain boundaries since such rotations may also be caused by local surface deformation or compatibility strains. The EBSD approach provided relative orientation information with a precision of approximately 0.5°.

The misorientation angle files from all the boundaries adjacent to voids were merged together to enable an overall comparison of voided and unvoided line segments. Pole figures and orientation distribution functions generated from these data sets revealed a correlation between voiding, heat treatment, and the degree of $\langle 111 \rangle$ texture, as detailed in Ref. 3. Briefly, weaker $\langle 111 \rangle$ texture was correlated with type 1 samples as well as the local microstructure surrounding voids.

Histograms of grain boundary misorientation angles, also called Mackenzie plots,[8] were created for the type 1 and type 2 samples and are shown in Fig. 1. For this study, we define low misorientation angles as those with $\leqslant$°12 misorientation. The percentages of low angle boundaries are 3% and 12% for type 1 and type 2 samples, respectively, as shown in Fig. 1(a). In addition to differences resulting from heat treatment, the fraction of low misorientation angle boundaries also varied as a function of location for the type 2 samples. Specifically, 18% low misorientation angle boundaries were observed at unvoided regions, compared to 2.5% at voided regions, as shown in Fig. 1(b). Little difference in this percentage was revealed for the lower reliability, type 1 voided and unvoided regions.

Misorientation angles of 60° may include $\Sigma 3$ twin boundaries since the film was preferentially $\langle 111 \rangle$. However, since twin boundaries generally exhibit low diffusivity and comparable twin densities were observed in the FIB images of both sample types, these boundaries are not expected to significantly affect voiding. The misorientation angle plots also did not reveal a correlation between 60° misorientations and voiding.

Implications about relative boundary diffusivities can now be made. Wardle *et al.*[9] derived the following relationship for the relative misorientation dependence of the grain boundary diffusion coefficient in low angle tilt boundaries, for misorientation angles less than or approximately equal to 10°:

$$\ln\left(\frac{D_{b,1}}{D_{b,2}}\right)=\frac{\theta_1}{\theta_2}. \tag{2}$$

$D_{b,1}$ and $D_{b,2}$ are the grain boundary diffusivities resulting from misorientation angles θ_1 and θ_2. The most drastic increase in diffusivity with misorientation angle occurs in this low angle regime. We assume that similar relative effects would be seen for boundaries of pure twist or mixed character. Note that larger misorientation angles are associated with higher boundary diffusivities. In addition, for large misorientations, boundaries begin to lose the anisotropic properties typically attributed to the dislocation networks found in low angle boundaries.[10] For the case of random high angle boundaries, diffusivities and energies are higher still since they are much more disordered and therefore contain more free volume.[11,12] There is also experimental evidence[11] for the presence of cusps of lower diffusivity at certain high misorientation angles, which may correspond to special coincidence boundary structures. However, our measurements did not reveal a significant proportion of coincidence relationships in these samples, and so we expect a correspondingly negligible contribution to voiding behavior. That type 2 samples voided less than type 1 samples is now further explained, then, since type 2 lines are comprised of a higher proportion of low diffusivity pathways, based on boundary misorientation angles. Further, those regions that underwent voiding in a given line were comprised of a *locally* higher proportion of high diffusivity boundaries than regions which remained intact. So, the local variation in grain boundary structure within a single line becomes significant in that sites for preferred void formation and growth can be present within a line.

Grain boundary structure affects not only void growth kinetics, but also void nucleation. Consider how misorientation angle is correlated to the nucleation energy for a grain boundary void. Kaneko *et al.*[13] determined the critical free energy change for the formation of a slitlike void in aluminum lines. They showed that the critical free energy change for void formation decreases proportionally with increasing grain boundary energy, independent of void geometry. The decrease is due to the fact that total grain boundary area decreases upon void growth. Since grain boundary energy increases with boundary misorientation angle,[11] the critical free energy change for void formation will also decrease with increasing misorientation angle. We expect therefore that void nucleation is preferred at higher angle boundaries.

Clearly, other factors involved in grain boundary structure will also play a role in both void nucleation and growth, including relative degrees of tilt and twist character, as discussed in a separate letter.[3] Further, the local occurrence of *groups* of adjacent high diffusivity boundaries may also play a role in controlling void nucleation and growth. These must all be considered in putting together a more complete picture of the stress voiding phenomenon.

In conclusion, this work and that described in Ref. 3 represent the first local analysis of film texture and grain boundary misorientations as they relate to stress voiding damage in copper interconnects. Improved stress voiding resistance following heat treatment was explained in terms of changes in grain boundary structure; the fraction of low misorientation angle grain boundaries altered the grain boundary energy and diffusivity. Grain boundary structure was shown to influence both the nucleation and growth of voids, illustrating the importance of local microstructure on stress voiding reliability.

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